The Use of Electrochemical Impedance Spectroscopy to Monitor Delaminations in Polymer Matrix Composites: A Review

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***Abstract*-A review on the use of electrochemical impedance spectroscopy (EIS) was performed to determine the applicability of using this method as another method of non-destructive evaluation on polymer matrix composites. The motivation of this review is to display that EIS can be a quicker, more reliable, and quantitative method for use on a class of materials which has become increasingly more utilized in high profile applications. Conductive fiber composites comprise the bulk of the study as the traditional EIS methods correlated nicely as one constituent of the measurement setup must be conductive. For this class of material, several delamination mechanisms were studied including: fatigue, absorption of solutions, and application of overpotentials, galvanic coupling, and microbial attack. Also, non-conductive polymer matrix composites were also studied via a two electrode EIS technique. It was determined that EIS is an applicable method for determining delaminations from degradation stimuli. Also, in some instances the EIS response could be an indication to the extent of influence on mechanical properties.**

Keywords-Polymer matrix composites; Non-destructive evaluation; Electrochemical impedance spectroscopy; Delaminations; Carbon fiber reinforced composites

I. INTRODUCTION

Advanced polymer matrix composites (APMCs) have become increasingly popular in military and aerospace applications due to their low weight and high specific properties. APMCs are typically defined by the incorporation of long, high strength fibers (carbon, aramid, etc.) in an advanced matrix material (epoxies, phenolic, etc.). The combination of these two phases gives the manufactured composite superior mechanical and thermal properties.[1] In the 1970's and 1980's proposed designs of the AV-8B aircraft had 23.3% (by structural weight) epoxy matrix/graphite fiber in such critical areas as the fuselage and substructures of the wing.[2] The success these materials found eventually lead to use in the commercial sector of flight with the Airbus 340 and Boeing 777 which have 15% and 13% APMC materials by weight. [3] As APMCs move into the future, the uses in unmanned ground vehicles, unmanned air vehicles, and larger, more sophisticated commercial aircraft (i.e. Boeing 787) will be predominantly composed of composite materials.[1] The increased utilization of these materials comes not only from the weight savings and exceptional properties, but also the ease of manufacturing complex shapes as compared to monolithic metals. The emerging technologies of advanced composites manufacturing allow for these complex geometries to be manufactured with low void content, thus increasingly higher fiber volume fractions. [4]

APMCs' mechanical properties are highly dependent on establishing an intimate bond between the polymer matrix and fiber since the mechanical loading must be transferred from the matrix through the interface/interphase to the fiber.[5] However, once a composite is exposed to stimuli (mechanical forces, chemical reactions, fungal deterioration, etc.) the bond between fiber and matrix begins to become delaminated from one another and as the stimuli is repeated the delamination area augments.[6] A fairly similar phenomenon can be observed in the area of coated metals. Electrochemical impedance spectroscopy (EIS) is a widely accepted technique in the field of polymeric coatings for monitoring corrosion processes at the metal/coating interface.[7-8] Through the use of circuit modeling or simple spectra analysis, the delamination of coating from substrate can be observed over an exposure time from several types of external stimuli. [9] EIS can predict coating failure quite successfully before the visual signs can be interpreted. The quantitative measure is very useful when establishing guidelines of when failure can be assumed, and this can be done by monitoring the magnitude or complexity in shape of the Bode plots, or the onset of multiple time constants (i.e. another RC circuit in the circuit model) within the Nyquist plots. Another accepted approach of predicting coating failure is assessing the individual elements of circuit modeling over exposure time.[10] The quantitative nature of these measurements makes it an ideal candidate for non destructive evaluation (NDE) of polymer matrix composites as compared to that of qualitative measurements such as ultrasonic, thermography, acoustic emission, etc.

II. INVESTIGATING DELAMINATIONS IN CONDUCTING FIBER APMCS VIA ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

APMCs which contain carbon or graphite fiber in an insulating matrix have been studied to a higher degree than that of non-conductive fibers because the conductive fiber can act as the working electrode in the conventional three electrode EIS measurement, or if the fiber volume fraction is above the percolation threshold, the entire composite specimen may act as the working electrode. The latter is usually employed when dealing with the use of carbon fiber

reinforced plastics (CFRPs) for retrofitting concrete structures where EIS has been used to monitor delamination effects.[11-13] However, this subject will not be covered in this review article due to macro-scale delamination being monitored between the composite/concrete interface and not the matrix material and carbon fiber interface of the APMC.

With the APMCs, several modes of delamination progression have been studied over the past three decades which include: delamination by fatigue, delamination by solution absorption, delamination by overpotentials, delamination by galvanic coupling, and delamination by microbial matrix attack. For clarification, the following sections will be ordered by the degradation characteristic causing delamination and void creation.

A. Delamination Monitoring by EIS Measurements for Fatigued APMCs

The fatigue process in polymer matrix composites have been shown to create delaminations of fiber from matrix by several proposed mechanisms including fiber failure, fiber bridging, matrix cracking, etc.[14] In 1987, R.C. Glass, et al. examined the modeled double layer capacitance from the EIS measurement as an indicator of the extent of delamination of carbon fiber from an epoxy matrix after flexural fatigue was administered to the composite material.[15] The EIS measurements were carried out in typical three electrode technique with a saturated calomel electrode (SCE) acting as the reference electrode with a Luggin capillary probe. Carbon was utilized as the counter electrode with a deaerated sodium sulfate solution being the electrolyte solution. Investigation of the double layer capacitance after 10^5 fatigue cycles displayed significant decreases with respect to the original measurements. It was determined that this substantial change in measured capacitance could be accounted for by the corresponding decrease in the active electrode area caused by breakage of carbon fiber during fatigue. Also, flexural fatigue is a known method of increasing the absorption of solution in composite materials which would thus increase the respective capacitance which did not occur in this experimentation.[16] Both of these assumptions come from the established equation for capacitance displayed below.

$$C = \varepsilon_o \, \varepsilon_r \, A / d$$

Where ε_o is the permittivity of free space, ε_r is the dielectric constant, A is the active area, and d is the distance between the two conductive "plates". In this instance the area, A, would decrease due to fiber breakage causing a lower capacitance. However, if moisture is absorbed by the composite the dielectric constant would be of mixed permittivity which would be altered to a value between the polymer (2.1 – 12.2) and water (76.6 – 80.2) which would thus increase the capacitance.[17] This phenomenon was explained along with another interesting aspect that was observed after the initial fatigue testing was completed. It was detected that the measured capacitance from the EIS measurements continued to decrease even after the fatigue cycles were completed. This was attributed to the atmospheric moisture or electrolyte solution still present within the composite causing swelling of the polymeric matrix and continued fiber breakage of either partially damaged fiber or those fibers which have residual stresses from fatigue acting upon them again causing active area to decrease and therefore the measured capacitance. Further observation of the impedance spectra lead to the belief that fiber breakage was the main component of changes in capacitance since the resistance increased (i.e. shorter fiber and thus longer and less conductive pathways). From this work, it was discovered that fatigue caused several phenomenon to change the capacitance measured at the carbon fiber/epoxy matrix interface.

B. Delamination Monitoring by EIS Measurements for APMCs by Absorption of Solutions

The second focus of the article written by R.C. Glass et al. was to examine the effects of wet-dry immersion cycles of both distilled water and sodium sulfate.[15] The first EIS measurement was carried out in the as-received manner (if applicable), the second measurement was conducted after drying in an atmosphere of 140°C for 48 hours, the third measurement was performed after 30 days of immersion at room temperature (sodium sulfate) or 4 days at 100°C (distilled water), and the last measurement was conducted after repeating the procedure for the second measurement. Again it was observed that the capacitance decreased with immersion of both the distilled water and a sodium sulfate solution. However, after drying the composite sample and subjecting the sample to an EIS measurement, the capacitance had increased in almost every instance when compared to the EIS measurement after the first drying cycle. The results of the higher capacitance (in this instance) can be directly correlated to the delamination, microcracking and void formation caused in the composite sample during the drying cycle. These voids and delaminations make the access of testing electrolyte easier to the fibers causing increased area from an electrical bridging effect between fibers in a conductive solution. This claim is further reinforced as S.R. Taylor, et al. continued research in this area to determine that an unsized graphite fiber in epoxy would exhibit increases in capacitance when said fiber was exposed to a temperature of 90°C and immediately subjected to EIS measurements afterward.[18] The difference in coefficient of thermal expansions between the matrix and fiber caused areas of limited contact which when filled with testing solution caused an increase in the active area and thus the capacitance when compared to that of the unheated samples.

Lastly, a trend was observed between the capacitance and shear strength of the distilled water immersion samples. The results displayed the loss of shear strength of the carbon fiber containing composite samples correlate well qualitatively with the decrease in capacitance of the immersed samples. The correlation of the capacitance and shear strength can be observed in Figure 1. This further supports the postulation that the increase in weight (i.e. water absorption) may cause fiber breakage from polymeric swelling effects and therefore a decrease in mechanical properties.

Another solution was used by S.R. Taylor et al. in a study using different concentrations of sodium hydroxide solutions to produce hydroxyl (OH^-) ions for delamination creation in bismaleimide matrix/graphite fiber composites.[19] Hydroxyl ions were the proposed species that caused the delamination in

other studies exhibiting these composites to cathodic polarizations.

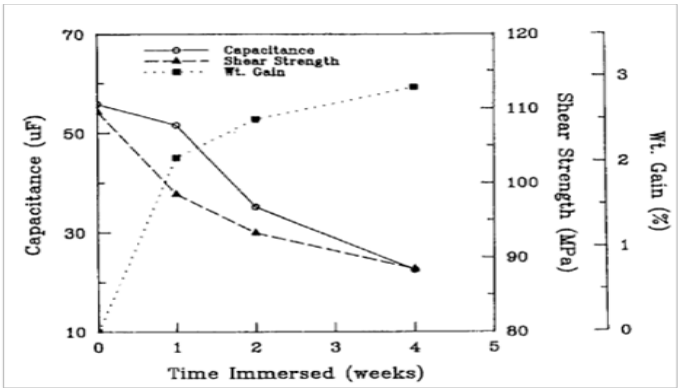

Figure 1 Relation of capacitance and weight gain to shear strength in immersion of composite sample in 90°C distilled water over exposure time

[Reproduced with kind permission from Springer Science+Business Media: Journal of Nondestructive Evaluation, Electrochemical impedance spectroscopy as a method to nondestructively monitor simulated in-service damage in a carbon fiber reinforced plastic, 6, 1987, 187, R.C. Glass, S.R. Taylor, G.L. Cahen, Jr., and G.E. Stoner,Figure 8.]

However, the results from the EIS measurements did not demonstrate a similar behavior to that of those exposed to cathodic polarization. The changes in phase angle plots for the caustic solution did not exhibit the behavior displayed by a "porous electrode" which can reveal the creation of delamination by changes in the phase angle over exposure time. The reason for this is that the dissolution of bismaleimide matrix was occurring in the bulk of the polymer, but not along the matrix/fiber interface which creates the voids and delaminations that can be measured by EIS. Also, if hydroxyl ions were being produced when impinged with cathodic potentials, the small pores and grooves would be imposed at the interface from the conducting species (i.e. graphite fiber) making it more likely to see the "porous electrode" effect.

C. Delamination Monitoring by EIS Measurements for Overpotentials

It is now well understood that carbon/graphite fiber with a glass cross weave polymer composites coupled with anodic acting metals creates a blistering effect at the surface of the composite material.[20-21] Blister formation is from evolving species created at the carbon fiber/polymer matrix interface during the electrochemical process induced by cathodic overpotentials. D. Kaushik et al. investigated the effect of applying cathodic overpotentials to carbon and glass fiber weave/vinyl ester matrix composites and monitoring these effects with EIS and the subsequent changes in modeled circuit elements.[22] The composite material was subjected to potentials of 0V, -0.65V, and -1.20V (vs. SCE) over various exposure times. To model this data, the proposed circuitry was initially established by F. Mansfeld and M.W. Kendig for coated metals that had exhibited the initiation of corrosion.[23-24] The models incorporated a two time constant circuit (i.e. two parallel resistor-capacitor circuits within the model) including solution resistance, R_s, "coating" capacitance, C_c, pore resistance, R_{po}, charge transfer resistance, R_{ct}, and a double layer capacitor, C_{dl}, or constant phase element. The main circuit element to monitor in this case, due to blister formation, was the pore resistance which could monitor the ease of electrolyte penetration. The results, in this instance, suggest that the electrochemical damage initiates at the carbon fiber/vinyl ester interface and moves outward toward the solution. This was proposed due to the resulting pore resistance values decreasing due to delamination and eventually void formation and these claims were reinforced by scanning electron micrographs. Also, pH measurements of the blisters were found to be very high due to the hydroxyl ions that were supposedly being produced within the composite structure. However, as mentioned previously S.R. Taylor et al., it is suggested that this hydroxyl ion was indeed formed, but may not be the key damaging species (the proposed damaging species will be covered later in this review article).[19]

M.N. Alias and R. Brown did a comparative study using a carbon and glass fiber weave in both epoxy and vinyl ester matrices, but the degradation procedure and analysis process were similar.[25-26] It was determined that the epoxy based composite experienced white deposit formation at the surface after varying lengths of cathodic potential exposure while the vinyl esters displayed blistering and black debris was found in the electrolyte solution. Circuit modeling of the EIS data suggested that a single resistor-capacitor circuit existed meaning the pore resistance was zero (i.e. a short circuit of electrolyte though the composite) for the epoxy composites at potentials less than -300 mV (vs. SCE). This could also be observed at the surface of the composites when more cathodic potentials than -300mV (vs. SCE) were applied to the samples. However, when the pore resistance was still detectable (i.e. voltage of -300 mV (vs. SCE) or more positive), it was determined that the values were higher in magnitude for the epoxy than the vinyl ester. Both composites exhibited damage dependent on the applied potential with more cathodic potentials causing greater damage by visual and electrochemical analysis. The impedance spectra displayed decreases in the magnitude with increased exposure time, and phase shifts (in frequency) toward lower frequencies with time. This work suggested possible degradation species at the -1200 mV (vs. SCE) to be the evolution of hydrogen to create the pores, or, due to the potential, direct reduction of the polymeric material.

A published article of S.R. Taylor's research in 1994 sought to determine the damage effects that occur when cathodic potentials and thus electrochemical processes are introduced at the matrix/fiber interface by looking at a the depth of penetration of a proposed "crevice" geometry between the matrix material and fiber.[27] This experimentation was conducted on a uni-directional bismaleimide matrix/graphite fiber composite which had EIS measurements conducted in typical three electrode fashion with the electrolyte being sodium chloride solution. Through extensive derivation, it was determined that the depth and width of the crevice could be monitored strictly by the phase angle which is measured during EIS experimentation. This derivation was originally proposed by de Levie to monitor the surface roughening of a scratch defect in electrodes for fast

electrochemical measurements.[28] Having graphite fiber act as one of the electrodes of the electrochemical system is beneficial since many of the underlying assumptions of the derivation are accurate due to its non-Faradaic region near the open circuit potential. Also, when surrounded in a polymeric matrix, a large portion of the impedance is due to the double layer capacitance observed when exposed to an electrolyte. Another advantage in using the phase angle to determine the penetration of delaminations is that the phase angle is an inherent parameter meaning it is independent of the area making this especially useful in laminate composites.

In order to create delamination effects, the author imposed cathodic potentials of -1.2V, -1.4V, and -1.73V (vs. SCE) in a 3.5% sodium chloride solution for varying amounts of exposure times. The potentials were maintained except when the specimens were being subjected to EIS experimentation which used the open circuit potential with a perturbation of 10 mV. The results of the penetration depth as a function of time for each voltage can be observed in Figure 2. As mentioned before, the most pertinent aspect of the impedance spectra was the phase angle which could be used in the series of equations derived to understand the depth of penetration of electrolyte (i.e. the length of delamination). It was observed that the larger overpotential caused greater lengths of delamination of the carbon fiber from the matrix material.

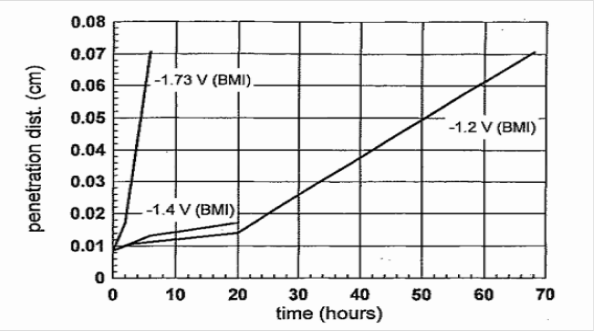

Figure 2 - Results of penetration depths of delamination calculated from the impedance phase angle data

[Reproduced by permission of The Electrochemical Society: Journal of The Electrochemical Society, The Detection and Analysis of Electrochemical Damage in Bismaleimide/Graphite Fiber Composites, 143, 1996, 451, S.R. Taylor, F.D. Wall, and G.L. Cahen, Jr., Figure 5.]

In order to calculate the penetration distance, several assumptions needed to be implied. For instance, the opening of the crevice (i.e. distance from fiber edge to polymer edge at surface) needed to be held constant even though this was known to change over time. Also, the value of the double layer capacitance was approximated from values suggested for the basal plane of graphite and crystal edges even though actual configuration was not determined during this study. Even with these assumptions, the extrapolated data to 12 months was very near to that found for actual data of epoxy/graphite fiber coupled with magnesium in seawater.

The findings through this experimentation were very beneficial for determining the delamination effects in graphite fiber composites when exposed to cathodic potentials which can be observed when these materials are coupled with common industrial metals in an electrolytic environment.

S.R. Taylor et al. continued this work to further explain the phenomenon observed involving cathodic potentials used with bismaleimide matrix/graphite fiber composites.[19] In this study, an understanding of the change in phase angle was studied in more detail. Since the phase angle is used to determine the depth of the delamination, the change in phase angles at corresponding frequencies can give better insight into the material's interfacial changes. Results revealed that with the more cathodic potentials the corresponding change in phase angle increases. Even though the -1.2 V and -1.4 V (vs. SCE) should be limited by diffusion processes, as defined by the potentiodynamic polarization data, the -1.4V results exhibited a larger change in the phase angle with increasing exposure time even though the imposed currents would be of the same magnitude and thus reactions at the interface should be similar. Further EIS experimentation was completed on samples that were exposed to smaller cathodic potentials ranging from -30 mV to -300 mV (vs. SCE). It was discovered that potentials ranging from 0 V to -100 mV (vs. SCE) exhibited phenomenon not observed in the other cathodic potentials, but once the -200 mV to -300 mV (vs. SCE) were measured the capacitance values again began to increase as expected. The explanation for the lower cathodic potentials behaving erratically was due to the composite sample's open-circuit potential varying up to 300 mV due to inhomogeneity in the sample and the sample's history. Also, the slight cathodic potential changes could create reduction of certain functional groups or electrostatic debonding at the interface between fiber and matrix. One of the most important findings of this work was the proposition of an alternative specie/species that cause the porosity to be created at the interface other than hydroxyl ions. From the change in phase angle results, a tail was observed at low frequencies in the cathodic potentials and that was not observed when hydroxyl radicals were readily present in solution immersion testing. This is believed to be caused by damage inherent from the generation of peroxide, superoxide radicals, and possibly hydroperoxyl radicals. Cleavage of the carbon-carbon bonds (amongst several other degradation mechanisms) within polymer matrices after reactions with oxy-radicals and peroxy-radicals has been well documented. [29-31] However, the degradation species have not been explicitly determined and is most likely to be different for each matrix material.

The application of anodic overpotentials was also investigated by S.R. Taylor et al.[19] Although the practical applications of having a very noble species such as graphite being subjected to an anodic overpotential is limited, it was suggested by the authors that this could indeed by the case if a stray current was administered to the composite. A large overpotential of +1.5 V (vs. SCE) was applied to the samples for 20 hours. Anodic polarization resulted in decrease in impedance magnitude with time, a shift of the capacitive region, in the impedance magnitude plots, toward lower frequencies, but very little depression in the amplitude of the phase angle peak. The shift in the impedance plots suggest the same activity that occurred in cathodic overpotentials, but the phase angle displayed little change in the low frequency phase angles, further reinforcing that the delamination or pore formations were not occurring.

D. Delamination Monitoring by EIS Measurements for Galvanic Couplings

Much like imposing cathodic potentials, coupling of carbon fiber composites with more active metals would produce similar effects in the presence of an electrolyte. S.R. Taylor also conducted experimentation to monitor the creation of delaminations when bismaleimide matrix/graphite fiber composites were coupled with aluminum, steel, copper, and titanium.[19] The results demonstrated similar, but not identical, trends observed when cathodic potentials were applied including impedances decreasing with time, the slope of the Nyquist plot decreasing, and phase angle measurements decreasing with increasing exposure time. As expected, the impedance changes were concurrent with the metal's electrochemical potentials in a sodium chloride containing solution with aluminum being most active at approximately -0.81 V (vs. SCE), next is low carbon steels at approximately -0.70 V (vs. SCE), then copper at approximately -0.20 V (vs. SCE), and lastly titanium at approximately -0.05 V (vs. SCE).[32] Again the phase angle decreasing with time was the main indicator that a "porous electrode" was being formed and the active area was increasing due to the formation of the peroxide radicals that degraded the polymer at the interface.

E. Delamination Monitoring by EIS Measurements during Microbial Attack

It has been shown that microbes tend to thrive off the additives (sizings, plasticizers, flame retardants, etc.) in carbon fiber reinforced composites, as it tends to be the site of microbe growth via scanning electron micrographs.[33] J.-D. Gu has provided research examining the relationship between growth of microbes at the carbon fiber/matrix interface using EIS as a technique to monitor the growth (and consequently the delamination of fiber from matrix) of these bio-organisms.[34-35] Graphite and glass fiber weave/epoxy matrix composites were manufactured and were then inoculated with a fungal consortium and then characterized by EIS and SEM after 179 days of incubation. EIS results displayed that the impedance magnitude was decreasing over exposure time while samples in non-microbe rich environments revealed increases after initial testing, but remained stable after initial testing. The shape of the impedance magnitudes exhibits both a decrease in the resistive and imaginary elements of the sample. This can be explained by both a decrease in the pore resistance (from microbe pathways) and an increase in the capacitive behavior due to increased active area which corresponds to a decrease in the imaginary impedance which is verified in the Nyquist plot. These observations were also visually verified using scanning electron micrographs.

III. INVESTIGATING DELAMINATIONS IN NON-CONDUCTING FIBER APMCS VIA ELECTROCHEMICAL IMPEDANCE SPECTROSCOPY

The use of a non-conducting fiber (i.e. glass, aramids, etc.) in an insulating matrix gives the overall composite an insulating nature. These materials are not necessarily suitable for electrochemical measurements to be performed upon them which is why very little research exists in this area. Nevertheless, the need for quantitative non-destructive measurement is still present as special-purpose fibers continue to be enhanced to the point of rivaling the strength and stiffness of carbon fibers. This enhancement of glass fibers has been observed in recent years with the development of several special purpose glasses including "S"-glass, "M"-glass, "C"-glass, etc.[36] Even though these fibers are not conductive, EIS can still be administered to the sample, but the use of a two-electrode technique must be used. In terms of non-conductive composites, the two electrode technique usually utilizes two electrodes of highly conductive metal. In this configuration, the working electrode (of the conventional three electrode set-up) will act as one of the metal electrodes and the counter and reference electrodes (of the conventional three electrode set-up) will be on the other metal electrode. A schematic representation of the two electrode technique is depicted in Figure 3.

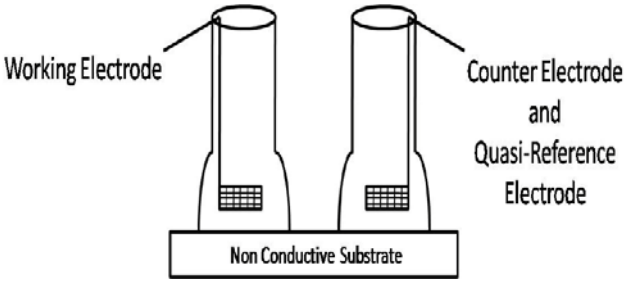

Figure 3 Schematic of the two electrode EIS setup

This technique has been shown to display similar trends to that of the three electrode set-up for coating systems which have undergone corrosion processes where point defects were not present.[37]

P.D. Fazzino, et al. researched the effects fatigue has on the impedance spectra for halogenated epoxy matrix/E-glass fiber composites.[38-39] By subjecting the composite material to fatigue mechanical loadings from cyclic compression, delaminations and voids were created. However, the first set of testing was to determine the effect immersion and drying would have on the overall electrochemical impedance spectra. By soaking the samples in water for 48 hours and then testing it intermittently while drying, it was determined that the spectra of the impedance magnitude changed from a resistor-capacitor circuitry to one dominated by the capacitive effect while drying. This could be explained by the inherent moisture of the soaked sample decreasing the resistance, thereby increasing the conductivity. Another preliminary study exhibited that the higher concentration of sodium chloride in solution decreased the impedance magnitude as the ion and electron movements were less restrained between the two electrodes. After initial studies, fatigued samples were then soaked in 3M sodium chloride solutions and measured in two electrode EIS fashion. A trend was observed explaining that with an increase in fatigue cycles a decrease in the impedance magnitude was observed. Figure 4 demonstrates the trend of decreasing impedance with increased fatigue cycles.

This is attributed to the phenomenon of void creation and delaminations causing increased solution uptake and thus less resistive pathways through the composite. Even for the

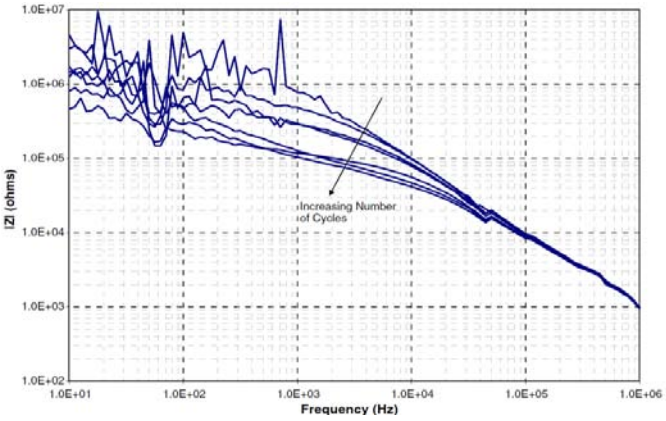

Figure 4 Decreasing impedance with increased fatigue cycles

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smallest amount of damage (roughly 20% of the cycles to failure), visual damage was unnoticeable, but detectable by EIS measurements.

Further work by this group investigated the direction of fatigue on the impedance results. It was determined that composite materials cut in the 0° and 90° (with respect to the warp fiber orientation) displayed similar results because a plain weave was selected and is quasi-isotropic while an off-axis bending samples (cut in the 45° direction) revealed drastically different results. The first noticeable trend was that the impedance magnitude was significantly lower (by over two orders of magnitude) in the 45°, and the high frequency impedance was changing while in the 0° and 90° orientations the high frequency stayed relatively similar. These changes in impedance spectra can be attributed to the 45° orientation bending causing more matrix driven behavior and thus more microcracking and void creation was occurring within the matrix. This causes an increase in the quantity of hydration sites and thus less resistive pathways to the ions and electrons causing a decrease in the resistive and capacitive behaviors of the composite. The large shifts in the high frequency impedance also suggest that the resistive and capacitive behavior is changing substantially. More extensive investigation examined the relationship between elements of the impedance spectra, area of damage, and strain to break in tension as a function of the fraction of life. It could be observed that trends did exist between the impedance magnitude at 1000 Hertz and initial slope of the Nyquist and the mechanical strain to break. Normalizing the data values against their initial values revealed that a correlation can be observed among the mechanical failure (strain to break) and electrochemical impedance spectra (initial slope of Nyquist plot) making EIS a suitable non-destructive evaluation technique for this material and mechanical loading.

IV. CONCLUSIONS

As advanced polymer matrix composites become increasingly popular in military, aerospace, and industrial applications, the need for quantitative non-destructive evaluation is becoming more apparent. This review article examined the applicability of using electrochemical impedance spectroscopy as a means to non-destructively evaluate this class of materials. Delaminations of both conductive and non-conductive fibers from an insulating matrix can be examined with this technique. With carbon/graphite fiber, equations have been developed to monitor the apparent characteristics of the fiber acting in a "porous electrode" manner, which can be used to quantify the length, and subsequently the volume, of delaminations. Studies have also been used to understand the key damaging species of coupling these carbon fiber composites with metals, and though an exact species has not been agreed upon, it is known that the species is dependent upon the potential applied and the type of matrix material involved. With the use of the two electrode technique and a hydrated sample, the delamination effects can be monitored with electrochemical impedance spectroscopy on non-conductive composites and correlation of the spectra characteristics with the mechanical properties can also be approximated. Overall, this technique of non-destructive evaluation is versatile and can give quantitative trends as opposed to many non-destructive techniques which tend to only be qualitative by nature.

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